AFML-TR-67-400

INVESTIGATION OF TECHNIQUES FOR THE INTRODUCTION OF LIQUID SAMPLES INTO A PLASMA ARC FOR ALLOY ANALYSIS

LOUIS E. OWEN

TOMORROW ENTERPRISES

TECHNICAL REPORT AFML-TR-67-400

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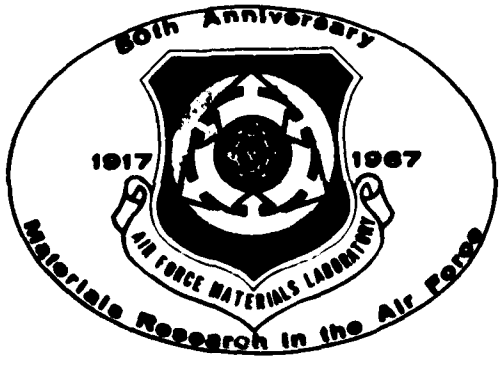
AIR FORCE MATERIALS LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO



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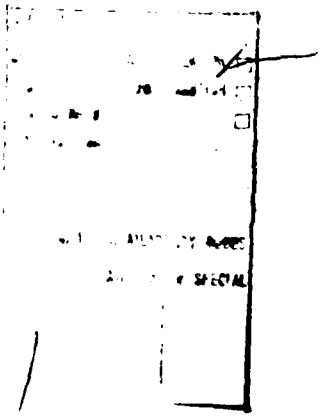

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FOREWORD

This report was prepared by TOMORROW Enterprises under USAF Contract No. F33(615)-67-C 1320. The contract was initiated under Project No. 7360, "The Chemistry and Physics of Materials," Task No. 736005, "Compositional, Atomic, and Molecular Analysis." The work was administered under the direction of the Air Force Materials Laboratory, Research and Technology Division, Air Force Systems Command, with Mr. James H. Muntz acting as project engineer.

This report summarizes the contracted six man-months of technical effort during the period December 1, 1966 to December 1, 1967. The manuscript was released by the author in November 1967.

The work at TOMORROW Enterprises was performed, or supervised, by Louis E. Owen, Principal Scientist and Co-owner. Experimental evaluation of prototype components and devices was accomplished during thirteen separate work visits, as a guest, at the National Bureau of Standards in the Spectrochemistry Section headed by Mr. Bourdon F. Scribner. Dr. Marvin Margoshes, of that section, furthered the project with specific technical advice and NBS liaison assistance.

This technical report has been reviewed and is approved.

Freeman F. Bentley

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Chief, Analytical Branch
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ABSTRACT

Ultrasonic nebulization is the basis of a sampling sub-system developed for the introduction of liquid samples into a plasma arc for alloy analysis. A focusing piezoelectric transducer at 1.4 megahertz creates a fine uniform aerosol from solution samples held in a chamber. Acoustical energy is water-coupled to this chamber through a thin plastic film. Ultrasonic nebulization provides an efficient transfer of analyte to the discharge zone of plasma arc excitation devices. Pneumatic and electrostatic nebulization, also investigated, are inferior to ultrasonic nebulization for general application. In particular, ultrasonic nebulization is practical with solutions far too concentrated for other techniques. The sample aerosol produced by the instrument developed has possible application to other instrumental analytical techniques such as atomic absorption and flame spectrophotometry.

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I. INTRODUCTION

Inefficient and irreproducible nebulization of liquid samples has been a limiting factor in the complete exploitation of plasma jet (arc) excitation in spectrochemical analysis. The original pneumatic nebulizers spraying the sample solution into the discharge zone of the jet failed to operate for extended periods with solutions containing more than 1 mg solids per ml. Dried residue from the sample spray deposits and invariably perturbs the gas stream and hence the nebulization process. This investigation hoped to improve the capability of pneumatic nebulizers or to adapt alternate techniques, specifically electrostatic or ultrasonic nebulization.

II. SUMMARY

The objectives of the original proposal which resulted in the contract of this report were partially attained in the contract period. Useful aerosols can now be made from sample solutions having salt concentrations much too great for nebulization by previously available techniques. It was not possible to achieve, during the contract period, "dry mist" aerosols with the ultrasonic nebulizer unit developed. The large gain in excitation efficacy attained, however, provides ample opportunity for eventual "trade off" of mass transfer rate for narrower droplet size distributions by means of auxiliary classifiers. More importantly, plasma jet devices excite these insonated aerosols more efficiently as reflected in increased sensitivity. The jet discharges also function gratifyingly quieter with the small droplet aerosols ultrasonically derived.

III. PNEUMATIC NEBULIZATION

Our original intention was to increase the efficacy of the pneumatic nebulizers commonly used in instrumental analysis by droplet size classification of their output. These nebulizers furnish a substantial mass transfer rate of liquid, but do so with a wide spread of droplet sizes. The larger droplets in the spray may carry a major portion of the solution volume transferred. Unfortunately, these large droplets seldom complete the necessary desolvation-vaporization-disassociation-excitation cycle during their brief transit through a plasma jet discharge. It seemed patent that small droplets would have a better chance to be excited at all, and that droplets of the same size would be more likely to exhibit excitation in the same zone of a discharge.

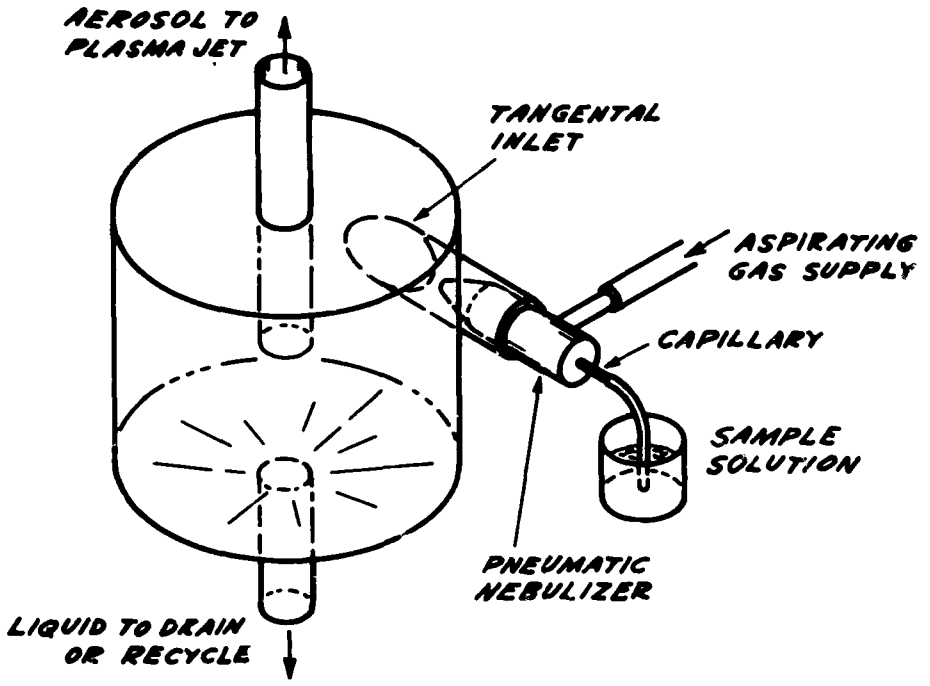

FIGURE 1 PNEUMATIC NEBULIZER-CLASSIFIER ASSEMBLY

We constructed many centrifugal classifiers of the general type shown in Figure 1. All of those fabricated extract 0.5% to 1.5% of the total solution volume from the output spray of pneumatic nebulizers in the form of a dry mist. The droplets in this mist are so small and uniform that they resist condensation or agglomeration. They do not deposit on glass surfaces they impinge and travel freely through ordinary tubing. Although the droplets of this dry mist excite efficiently in an electrical discharge. they have too low a mass transfer rate to compete with the inefficiently excited unclassified aerosols from which they are extracted. We unsuccessfully attempted to increase the droplet size distribution from classifiers to increase the mass transferred. Only drift tubes in which the distance of transport determines the size of carried droplets permitted inclusion of other than the finest droplets; but they seemed impractical for general use. The technique, however, is essentially involved in the ultrasonic instrument of final choice. In this system, the size of the largest droplets carried to the jet is not independent of the distance between the nebulization and discharge chambers.

Serious consideration was given to custom fabrication of better pneumatic nebulizers. Inherent problems of geometry and materials of construction limited this approach. Choosing corrosion resistant materials was no more than difficult but never became pertinent. The stumbling block of geometry is fundamental.

The primary disruptive force for aerosol production from pneumatic nebulizers comes from the difference in velocity between the driving gas stream and the liquid stream. The greater the differential velocity, the greater the efficiency (gas volume/liquid volume) and the smaller the droplets. Since the differential velocity is primarily a function of the gas velocity which follows the pressure drop across the gas exit orifice, this drop must be great for efficient aerosol formation with small droplets. But to reach the high pressure drop condition without concomitant excessive gas flow, requires tiny orifices which are suitable only for very dilute solutions. So while it is possible, as at the NBS, to design very efficient pneumatic nebulizers with small orifices, their analyte mass transfer rate remains less than for ordinary, less efficient, commercial pneumatic nebulizers. In dc arc plasma jet excitation great pneumatic nebulizer efficiency is not of such importance as to overshadow mass transfer capability.

IV. ELECTROSTATIC NEBULIZATION

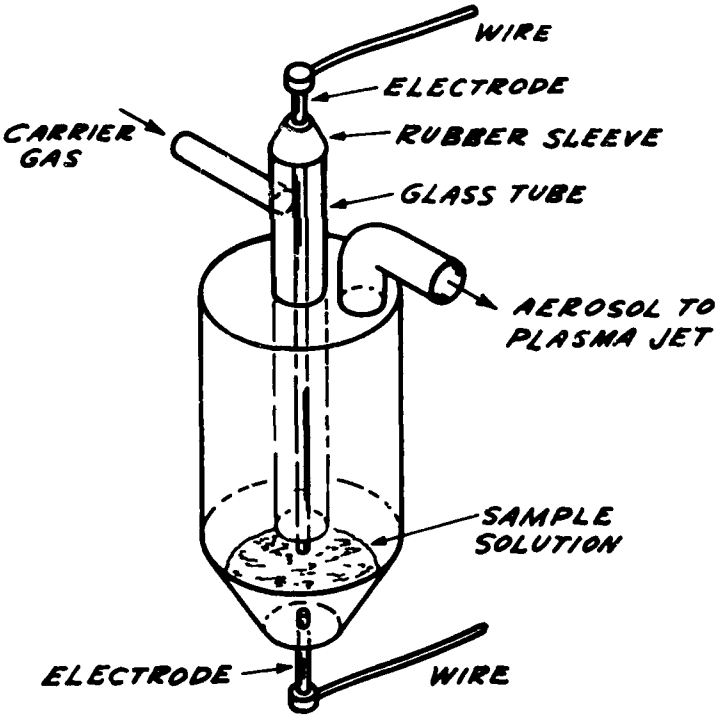

FIGURE 2 ELECTROSTATIC NEBULIZER

Our initial approach to electrostatic nebulization used the arrangement of Figure 2 in which a high voltage ac or dc spark directly impinged on the surface of an electrically conductive liquid sample. A 150 watt power supply furnished 8 kvac or 11 kvdc with or without capacitative shunts. Electrostatic nebulization demonstrated dependence on the physical and chemical properties of the solution as its most serious drawback. Furthermore the direct heating of the sample by the electrical sparks necessitates good cooling and temperature control of the solution. No evidence accumulated that the technique offers advantages commensurate with the complexity of the necessary instrumentation.

An attempt was also made to superimpose electrostatic augmentation on pneumatic nebulization. This technique also proved quite sensitive to solution properties such as viscosity and ionic concentration. Such sensitivity prevents application for a variety of solution types. A further limitation appeared when quite successful open-air experiments failed when transferred to enclosed chambers with different dynamic pressure conditions. The ultrasonic technique was by this time showing too much promise to justify continuing electrostatic tests.

V. ULTRASONIC NEBULIZATION

A. Submerged Nebulization Chamber

Our first tests of ultrasonic nebulization used an 800 kilohertz, flat, piezoelectric transducer in the assembly of Figure 3. The transducer was variably powered by a 100 watt, three-stage amplifier driven by a simple manually tuned oscillator. The cooling water served as an acoustical energy coupling medium and was intended both to cool the transducer (for frequency stabilization) and remove absorbed energy from the sample (for density stabilization). The basic configuration persisted through nearly a dozen prototypes functionally similar but resembling the original less and less.

An early alteration was the substitution of an intrinsically focusing piezoelectric transducer for the flat transducer and its accessory lens. The curved transducer eliminates loss of acoustical energy through absorption in the lens required by the flat transducer to increase the flux density. Then, too, particularly with an acrylic resin lens, the lens figure may deteriorate after prolonged exposure to an ultrasonic field. It was also advantageous to our project that the focusing transducer resonates at a 50% higher frequency than the flat transducer. Operation at higher frequencies shifts the droplet size distribution towards smaller diameters in any aerosol produced by ultrasonic bombardment.

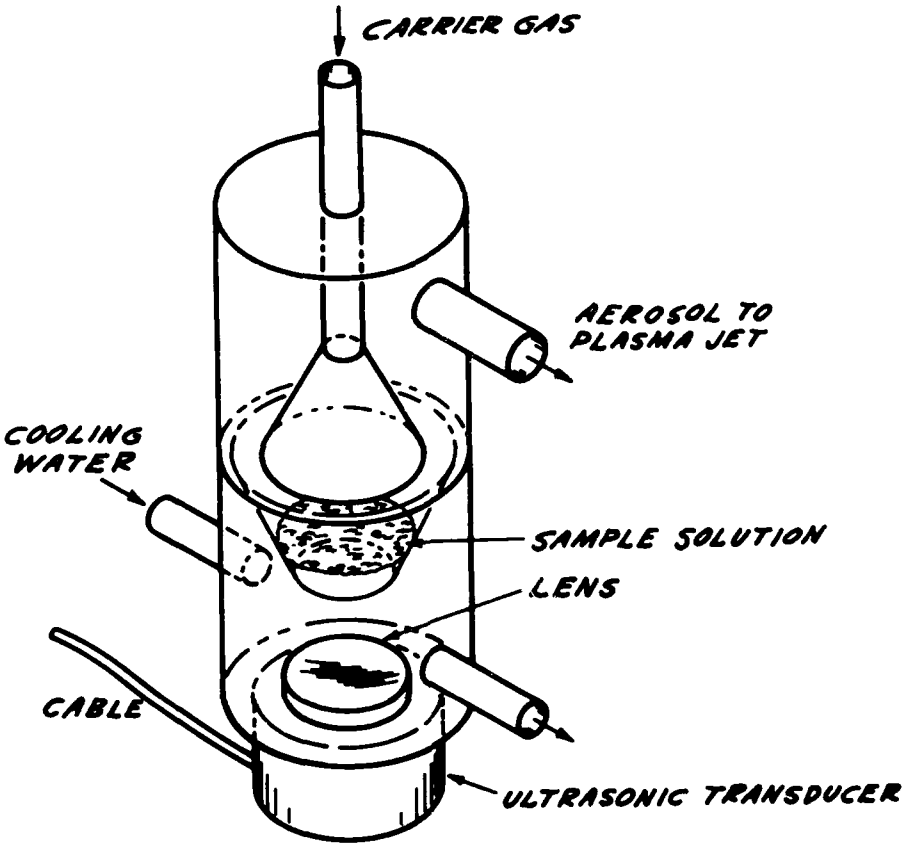

FIGURE 3 ULTRASONIC NEBULIZER WITH WATER COOLING

The second change, from the unit of Figure 3, brought into use a cylindrical sample solution chamber. This chamber, still separated from the cooling-coupling water by a thin plastic film, could be raised or lowered to zones of varying acoustical flux densities in the converging beam from the focusing transducer. In subsequent prototypes, the sample chamber was fitted with a complex array of connecting tubes. Some tubes served only for sample introduction and withdrawal while others tested various schemes for aerosol pickup and transport.

A large number of components for eliminating carryover of large droplets were fabricated and tested. As considerable splashing accompanies the "fountain" effect associated with high power density operation the carrier gas entrains some large drops as well as the desired aerosol disrupted by ultrasound from the sample solution. Even in the final model this phenomenon is not completely absent and deposited drops may be seen in the tubing between the nebulizer and the plasma jet.

One fortunate aspect of ultrasonic nebulization was manifest early in the study. The technique proved well suited to the nebulization of solutions too concentrated for routine pneumatic nebulization. It is obviously desirable that any nebulization technique be operable with solutions having high original sample to solvent ratios. At the time it was decided to limit additional work to the ultrasonic technique, useful aerosols had already been produced from a solution containing 30 grams of NaCl per 100 ml.

B. Non-submerged Chamber

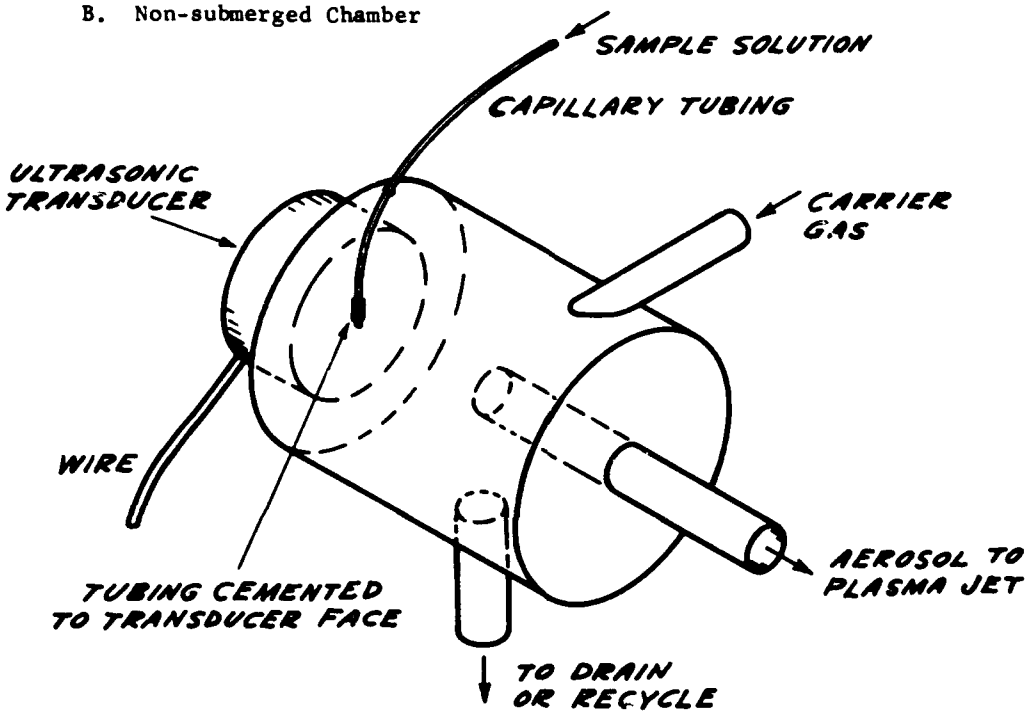

FIGURE 4 ULTRASONIC NEBULIZER-DIRECT IMPINGEMENT

The device of Figure 4 was suggested in the original contract proposal as potentially capable of "flash" nebulization of liquids fed continuously to it. Such a unit should be adaptable both to small sample volumes and to automated sample handlers with gas bubbles interposing discrete sample solutions. However, the device requires a piezoelectric crystal with a chemically resistant coat over its electrode surface. The coating is subject not only to the corrosive action of the sample solutions but simultaneously must withstand the huge accelerations of high frequency excursions. Such coatings were not available during the contract period for transducers of suitable geometry. A recent announcement (1) of a disc transducer protected by a "glass-like structure" suggests that the arrangement may yet

(1) Macrosonics Corporation, Rahway, N. J. Undated press release on their new Type A-804 Glazed Ceramic Transducer.

be practical. Sales literature identifies the facing only as a special ceramic which allows surface glazing "inert to most chemicals."

We sidestepped the temporarily restrictive requirement for a corrosion resistant transducer by water coupling to a solution interface of a metal or plastic film (Figure 5). The film vibrates much as the transducer face, though less forcibly, and sample liquids on its surface are disruptively repelled with extensive nebulization.

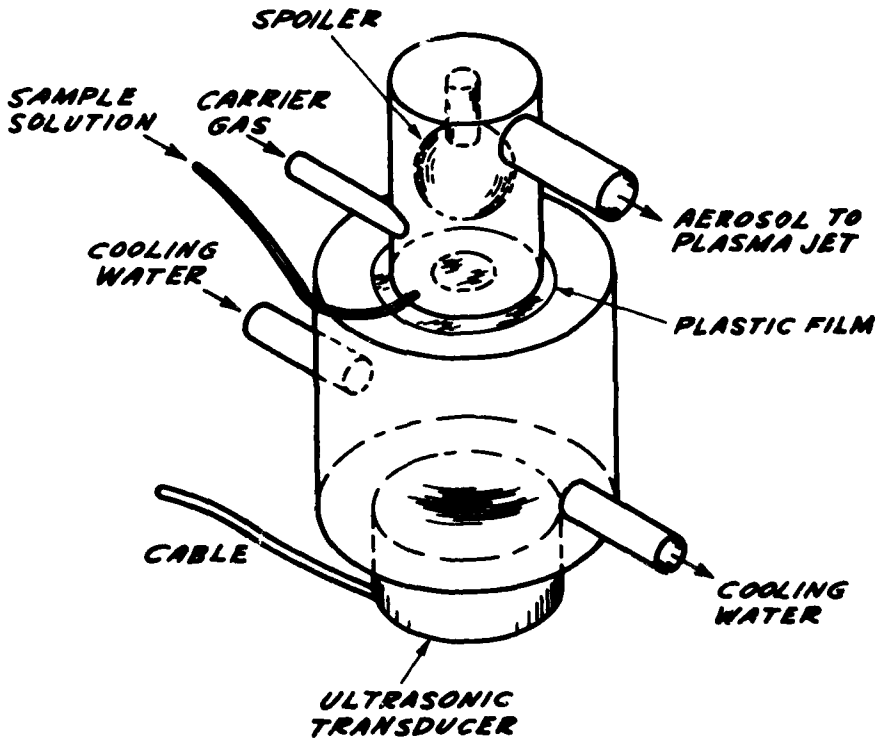

FIGURE 5 ULTRASONIC NEBULIZER-DISPLACED IMPINGEMENT POINT

While this configuration was retained for the final prototype developed, its use for small sample volumes and continuous operation was not completely established. Under the time pressure of the contract deadline, it was necessary to choose a more immediately available approach (batch process) for building the deliverable sampling sub-system. We are left vaguely troubled, however, with the belief that "flash" nebulization on ultrasonically vibrating surfaces is an exploitable technique as others maintain (2).

C. Nebulizing Device Developed

Performance evaluations of non-archival value were regularly made on the various ultrasonic nebulizer assemblies prototyped. Measurements obtained with photographic spectrographs or a photoelectric spectrometer at the NBS were used in guiding or justifying design modifications. Principally involved was the device of Figure 5 from which an improved apparatus was derived. We kept the non-submerged nebulizing chamber not with continuous feed, but, with batch samples of 5 ml transferred at one time to the chamber. When the sample is in the chamber, it may be insonated there and the unnebulized portion discarded after an excitation.

It is necessary to prevent contamination between successive samples while the vertical walls of the nebulizing chamber retain some spattered drops after withdrawal of the unnebulized solution. Extensive efforts to provide in-place rinsing of the entire nebulizing chamber foundered and we split the section. The upper part of the nebulizing chamber now plugs into a plastic base in which all sample manipulations except aerosol withdrawal are executed. The upper chamber part must be replaced between samples. It is reusable after rinsing and drying.

1. Construction Details

The basic nebulizer assembly, Figure 6, is driven by a curved piezoelectric crystal mounted in a brass case (3). The transducer subassembly is held by brass screws to a water chamber fabricated of acrylic resin pieces. The top surface of this chamber is 69.5 mm above the center of the 47.5 mm diameter piezoelectric element which is curved on a 95 mm radius. An opening in the top plate of the water chamber is edged by a partly inletted O-ring, nominally 3/4" id 1/16" w, which seals a plastic film against the bottom of the nebulizing chamber proper. If the film were stretched tight (not a specific requirement) it would be 70 mm above the surface of the piezo element at its center. The plastic film found generally suitable is MYLAR at 0.00025" thickness or, when this plastic is chemically vulnerable, FEP film at 0.0005" may be used. The FEP film has less tensile strength and exhibits non-elastic extension when stressed but it is resistant to a wide variety of solutions.

(2) Kirstein, W., Bertilsson, G., Anal. Chem. 38, 648 (1966)

(3) The DeVilbiss Company, Somerset, Pennsylvania
Transducer Assembly 8-87, Part Code 41732 501

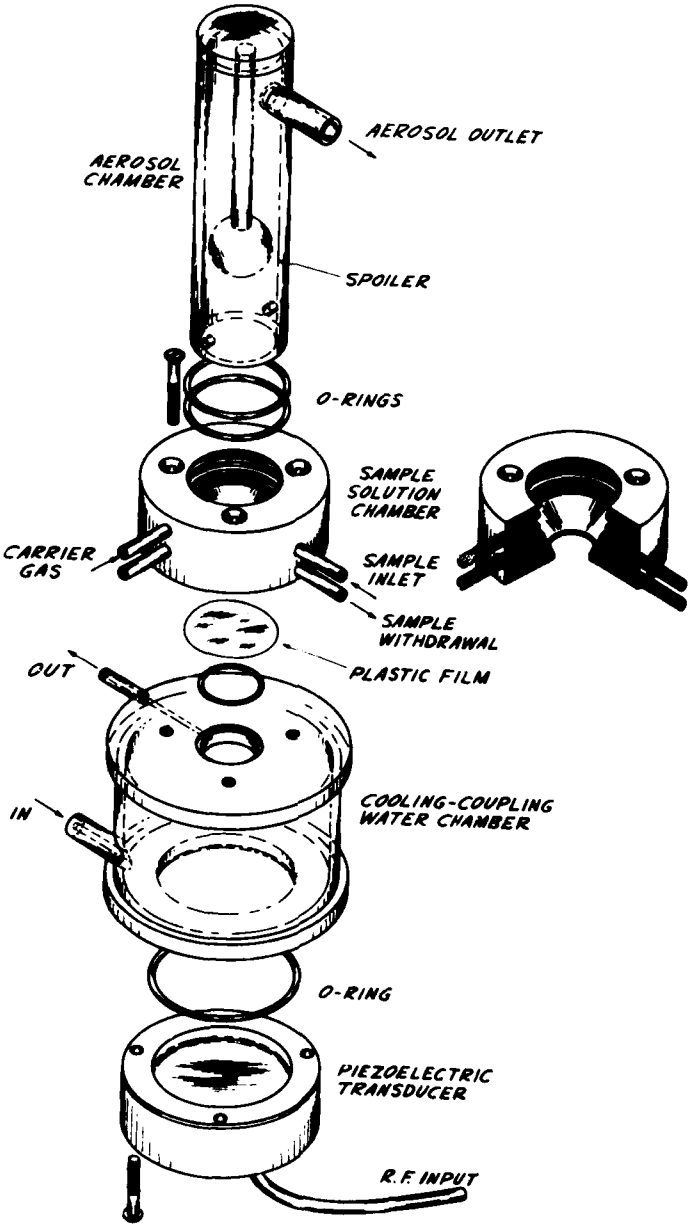

FIGURE 6 ULTRASONIC NEBULIZER ASSEMBLY

The plastic film presses against the bottom section of the nebulizing chamber and forms the floor of the sample section volume. The piece is plastic, 64 mm in diameter and 24 mm thick. The solution volume is an inverted truncated cone, 16 mm diameter at the film and 28 mm diameter up 12 mm from the film. The upper 12 mm of the piece is machined to accept, as a plug-in, the cylindrical aerosol-withdrawal section of the nebulizing chamber. VITON O-rings, nominally 1½" id 3/32"w, facilitate the replacement of the top section. The annular space between the O-rings is part of the inlet system for the carrier gas into the aerosol chamber.

In the solution section of the nebulizing chamber, separate connections permit injection and withdrawal of the sample solution, injection of rinse water, and admittance of the carrier gas as mentioned above.

The aerosol section of the nebulizing chamber is a plastic cylinder, 26 mm id x 32 mm od, 16 cm high. Its dimensions, as all others given, except possibly for the transducer film separation, are not known to be especially pertinent. The film-transducer distance matters because the energy from the element converges as it approaches the film. The power density flux therefore is increasing. As the film itself absorbs some of the acoustical energy, it will deteriorate and perforate, in time, when some critical flux is exceeded. Our experience showed that fin films are much more power tolerant, presumably because of more efficient cooling.

a. RF Power Generator

The rf power generator for driving the ultrasonic transducer can be any unit capable of furnishing about 100 volts at 1.3 to 1.4 megahertz with a power level of 25 to 100 watts. Frequency adjustment provision is required as the piezoelectric element is affected by its mechanical loading. TE tested a number of power generators including multi-stage supplies. For most of the contract period, we relied upon a one tube oscillator from commercially available equipment (4). This is the generator circuit, Figure 7, also included in the final instrument package.

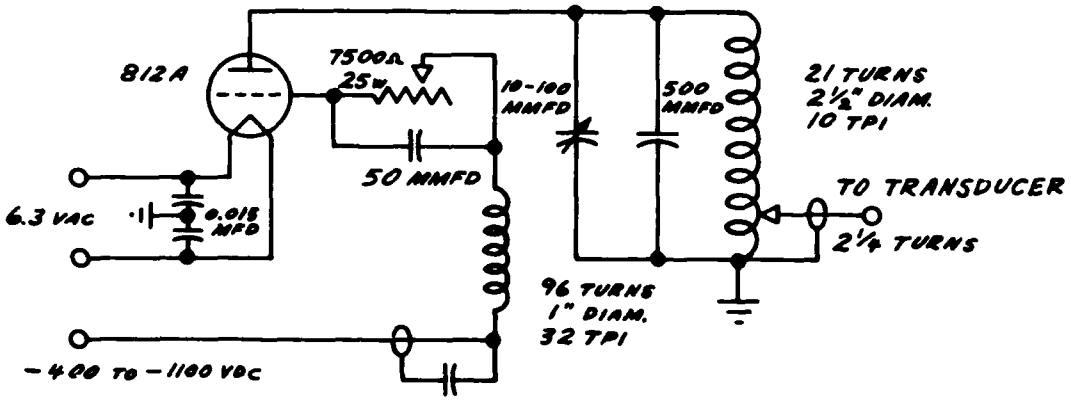

FIGURE 7 RF GENERATOR

(4) The DeVilbiss Company Model 800-882 Ultrasonic Generator

2. Operation

Preliminary design information and analytical data were obtained from the basic ultrasonic nebulizer unit set up in breadboard fashion. Acquiring adequate application information, even for research analyses, requires that the basic unit be integrated with ancillary equipment simplifying its operation. We assembled, for the Air Force development prototype, an instrument package aimed for the operating scheme of Figure 8.

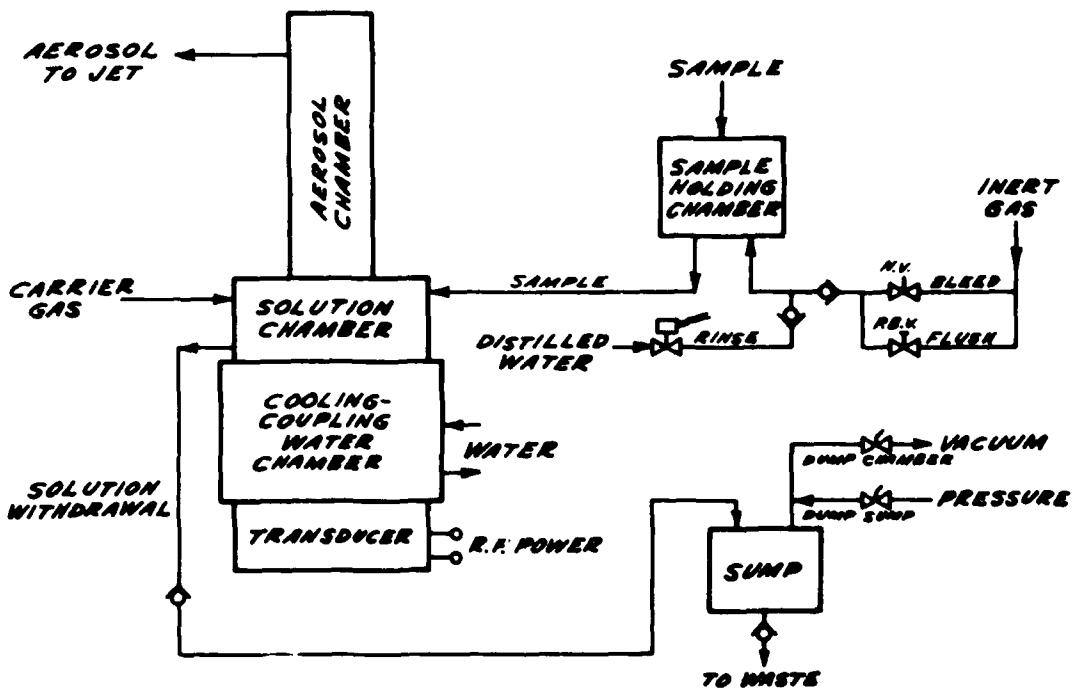

FIGURE 8 ULTRASONIC NEBULIZER - OPERATING SCHEME

A five milliliter portion of the sample solution is pipetted into a small sample holding chamber and the chamber is stoppered. Inert gas at a low flow rate displaces the sample solution from the holding chamber and drives it into the solution volume of the nebulizing unit. Once the jet is started, carrier gas flows into the nebulizer and on through to the sample inlet of the jet. The gas stream does not have a sample aerosol to carry, however, until the rf generator is powered and the transducer driven. The start command for this nebulization could be coordinated with the controls of the spectrometer detection system or spectrograph shutter.

The sample solution in the nebulizing chamber rests on the plastic film which is slightly concave as a consequence of less than atmospheric pressure in the water chamber below it. The pressure condition is easily maintained by limiting the water inflow while using an open-ended, gravity drain tube.

When an excitation is completed, a slight vacuum drawn on the sump chamber results in the unnebulized sample solution being forced into it. The used aerosol chamber is replaced with a clean dry one. The solution chamber rinses with distilled water under manual control. Provision is also made for rinsing the sample holding chamber.

Any time the sump chamber is nearly full, it may be dumped to a waste drain by pressurizing. This action could be initiated from electrical contacts built into the sump chamber.

3. Cross-Contamination

Experiments made during the final days of the contract provide hope that the entire aerosol chamber may not require replacement between samples to avoid cross-contamination. It may be sufficient to change the cap section containing the exit orifice and the suspended plastic ball. Tests of the flushing system for the solution chamber and sample holding chamber proved that manual flushing with a squeeze bottle was more reliable than a fixed geometry, automatic system. With manual washing, more of the aerosol chamber can be left in place for flushing between samples. The sample withdrawal system makes the manual operation relatively painless.

In one test, after nebulizing a sample of ten grams of steel dissolved in sixty milliliters of solution, we rinsed the chambers manually with the wash bottle. The Fe detection sensitivity was then increased by a factor of one million and deionized water was nebulized. The presence of iron was not detected.

VI. SPECIAL EQUIPMENT

A. Pumps

At the start of the contract period we were convinced that precise operation of any successful solution nebulizing technique would require removal of feed rate control from the device itself. Force feeding with independent control seemed imperative to minds well acquainted with the problems of pneumatic nebulizers. It was our opinion that a low flow rate pump capable of handling corrosive solutions would be needed. Nonpulsating flow seemed an additional requirement, especially for the pneumatic nebulizers on which early work was concentrated. A number of pumps were purchased and others evaluated. None were even partially satisfactory; pulsating flow being prevalent in spite of advertising claims to the contrary. TE subsequently undertook to develop a better pump. It was not the most successful of our efforts.

Common shortcomings of low flow pumps such as their inadequate power and control, poor fittings and non-resistant materials of construction are easily overcome. Eliminating pulsations is another matter. With tubing pumps, such as we built, pulsation frequency is increased and amplitude is attenuated with higher speed operation. Combining the flow from out-of-phase tubes is also helpful. These points were established but nonpulsating operation became less germane as pneumatic and electrostatic techniques were shelved; so were our pumps.

Gas displacement devices, as used for sample transfer in the system finally developed, were considered for positive feeding in all of the nebulizing systems but not very seriously, while we emphasized continuous feed. Displacement is a technique more suited to batch operation. It is likely, however, that a continuing study would include an evaluation of pressure drives for nebulizing chambers optimized for small sample systems.

B. Desolvation Apparatus

It was thought at first that aerosol desolvation external to the plasma jet would provide an important improvement in sample-to-carrier gas ratios. This improvement, if real, should be independent of the particular nebulization technique employed. The gain in sample to carrier gas ratio is, of course, only manifest if the solvent occupies an appreciable fraction of the aerosol volume. In practice this is usually trivial except for very volatile solvents.

External desolvation can be important when the very presence of the solvent in the working zone, of an arc, flame, or plasma jet, is deleterious to the desired action there. Flames are cooled and rf plasmas are severely perturbed by the presence of water in particular. For dc arc plasma jet excitation, surprisingly, we found desolvated aerosols are contraindicated. The phenomenon was not carefully investigated, as it should be, but no immediate gain was apparent. Rather, the desolvated aerosol seemed to produce a slightly less stable plasma discharge.

VII. CONCLUSIONS

An ultrasonic nebulization, sampling sub-system for the introduction of liquid samples into a plasma jet for alloy analysis has been reduced to hardware. This is the positive aspect of studies which also determined that we could not appreciably improve pneumatic nebulization and that electrostatic nebulization is more suited to specific than to general application. The ultrasonic instrument, in retrospect, would have benefited from more extensive development effort though, properly, not at the expense of investigations into pneumatic and electrostatic techniques.

The sampling sub-system developed did not exist a year ago, is better than TE could have assembled in September 1967, and is not as good as we will assemble some day. It represents our best knowledge and ability at this time. Encouraged by the support of this Air Force funding and intrigued by the remaining challenges of the problem, TE will continue to modify, improve, and evaluate ultrasonic sampling techniques. Specific instrumentation will be refined for plasma jet excitation and adapted for flame excitation and atomic absorption spectrophotometry.

VIII. RECOMMENDATIONS FOR FUTURE WORK

This development contract resulted, as all investigations tend to, in the raising of more questions than it answered. As a rather modest effort (funded for six man-months of technical work) it may have been especially frustrating in ruling out the possibility of rigorously exploring difficult areas of promise. For instance, we believe that small diameter nebulizing chambers, properly impedance matched with low power ultrasonic generators, might enable aerosol production from small sample volumes. In conjunction with small diameter, low power units, higher frequency insonation seems worthwhile. TE is still awaiting a custom transducer resonating, in air, at 5 megahertz. We predict aerosols with droplet size distribution shifted sharply to smaller diameters.

The direct impingement method also deserves more extensive study with ceramic-coated, piezoelectric elements resistant to corrosive solutions. While none of such elements acoustically couple appreciably with gases, preliminary work showed that liquids do nebulize well directly from their vibrating surfaces. The required driving power should be very low for this technique and simpler instrumentation may be possible.

Future work is definitely needed to increase the routine operational ease of the sampling system developed. Prevention of cross-contamination between samples must be strengthened and simplified. The operator should be relieved of all manipulations beyond presenting the sample to the equipment. More complete utilization will require compatability with automated accessories, especially those for sample input.

IX. TECHNICAL PRESENTATIONS

"Nebulization of Analytical Solutions" March 21, 1967 at Dayton, Ohio, at the All-Day Meeting of the Ohio Valley Section of the Society for Applied Spectroscopy.

"The Nebulization of Liquid Samples For Use in Instrumental Analytical Techniques" April 21, 1967 at Dayton, Ohio, at the 76th Annual Meeting of the Ohio Academy of Science held at the University of Dayton.

"The Nebulization of Analytical Solutions for Instrumental Techniques" October 11, 1967 at Gatlinburg, Tennessee, at the 11th Conference on Analytical Chemistry in Nuclear Technology sponsored by the Oak Ridge National Laboratory.

Pending: Informal presentation at National Bureau of Standards concerning the application of this project to their work.

"Ultrasonic Nebulization of Solutions for Instrumental Techniques" proferred to 1968 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy.

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13 ABSTRACT

Ultrasonic nebulization is the basis of a sampling sub-system developed for the introduction of liquid samples into a plasma arc for alloy analysis. A focusing piezoelectric transducer at 1.4 MH creates a fine uniform aerosol from solution samples held in a chamber. Acoustical energy is water-coupled to this chamber through a thin plastic film. Ultrasonic nebulization provides an efficient transfer of analyte to the discharge zone of plasma arc excitation devices. Pneumatic and electrostatic nebulization, also investigated, are inferior to ultrasonic nebulization for general application. In particular, ultrasonic nebulization is practical with solutions far too concentrated for other techniques. The sample aerosol produced by the instrument developed has possible application to other instrumental analytical techniaues such as atomic absorption and flame spectrophotometry.

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